

In re application of Docket No:Q84708
Arase, et al.

Filed : June 16, 2003

Title : PROCESS FOR PRODUCING

FLUOROCOPOLYMER , FLUOROCOPOLYMER , AND MOLDED OBJECT

Honorable Commissioner of Patents and Trademarks,
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I, Tadaharu Isaka, a citizen of Japan and having postal mailing address of c/o DAIKIN INDUSTRIES, Ltd., Yodogawa Plant. 1-1, Nishihitotsuya, Settsu-shi, Osaka 566-8585 JAPAN, declare and say that:

In March 2001, I was graduated from Hokkaido University and received a Master Degree in Science;

I am one of the inventors of the above-identified application and familiar with the subject matter thereof;

I have read the Official Action mailed and the references cited therein and I am familiar with the subject matter thereof;

I respectfully submit herewith my exact report thereon.

The following experiments were conducted by me or

under my direct supervision.

I produced the fluorocopolymer by the method for producing a fluorocopolymer which satisfies requirement of the present invention and the fluorocopolymer by the method for producing a fluorocopolymer which does not satisfy it, examined their property and compared to the present invention.

In Example 2,4 and Comparative Examples A,B, the differential scanning colorimetric measurements were carried out under the following conditions.

Measuring apparatus: Shimadzu DSC-50

Temperature programming: 10°C/min

Measurement temperature range: 130 to 350°C

Sample size: About 10 mg.

Comparative Example A

Production of a fluorocopolymer without additional feeding of PSFS

A 500-mL SUS stainless steel autoclave was charged with 266.4 g of perfluorohexane as a solvent, and 166.0 g of PFSF, followed by deaeration. With stirring at 800 rpm and at a temperature of 30°C, TFE was charged into the autoclave under pressure to a total pressure of 0.38 MPa and, then, 3.56 g of a 8% (by mass) solution of the initiator di(ω -hydroperfluorohexanoyl) peroxide in perfluorohexane was fed under pressure to initiate the polymerization reaction. During the reaction, TFE was introduced from outside the system to maintain the

pressure at a constant level. After the lapse of 2.3 hours, the unreacted TFE was discharged out of the system and the polymerization reaction was thus terminated. The stirring condition within the system was good. After completion of the polymerization reaction, 250 mL of chloroform was added, followed by 30 minutes of stirring. Then, solid-liquid separation was effected using a centrifugal separator, and 250 mL of chloroform was added to the solid matter, followed by 30 minutes of stirring. This procedure was repeated three times to wash the polymer. Then, the thus-washed polymer was deprived of the residual chloroform under vacuum at 120°C to give 20.1 g of copolymer.

The fluorovinyl ether derivative content C in the thus-obtained copolymer as estimated by fused state NMR at 300°C was 14.9 mole percent, the MFR at 270°C under a load of 0.21 MPa was 10.7 g/10 minutes, and the fusion peak area in DSC as observed at about 320°C was 0.3 J/g.

Comparative Example B

Production of a fluorocopolymer without additional feeding of PSFS

A 100-mL SUS stainless steel autoclave was charged with 43 g of perfluorocyclobutane as a solvent, and 11.4 g of PFSF, followed by deaeration. With stirring at 1.000 rpm and at a temperature of 30°C, TFE was charged into the autoclave under pressure to a total pressure of 0.71 MPa and, then, 1.0 g of a 8% (by mass) solution of the initiator di(ω -hydroperfluorohexanoyl) peroxide in

perfluorohexane was fed under pressure to initiate the polymerization reaction. During the reaction, TFE was introduced from outside the system to maintain the pressure at a constant level. After the lapse of 3.2 hours, the unreacted TFE was discharged out of the system and the polymerization reaction was thus terminated. The stirring condition within the system was good. After completion of the polymerization reaction, 250 mL of chloroform was added, followed by 30 minutes of stirring. Then, solid-liquid separation was effected using a centrifugal separator, and 250 mL of chloroform was added to the solid matter, followed by 30 minutes of stirring. This procedure was repeated three times to wash the polymer. Then, the thus-washed polymer was deprived of the residual chloroform under vacuum at 120°C to give 4.2 g of copolymer.

The fluorovinyl ether derivative content C in the thus-obtained copolymer as estimated by fused state NMR at 300°C was 5.3 mole percent, and the fusion peak area in DSC as observed at about 320°C was 4.1 J/g.

Results

The results obtained are shown in Table 1. The results obtained by Example 2 and 4 in the specification are also shown in Table 1.

Table 1

	Examples (in the specification)		Comparative Examples	
	2	4	A	B
Additional feeding of PSFS	With additional feeding		Without additional feeding	
C (mole percent)	15.9	5.5	14.9	5.3
MFR (g/10min)	14.4	—	10.7	—
Fusion peak area in DSC (J/g)	0	2.7	0.3	4.1

I obtained the fluorocopolymer its composition distribution and molecular weight distribution are minimized by the present invention.

The fluorocopolymer obtained by the comparative examples did not have so excellent distribution as the present invention.

I declare further that all statements made herein of my own knowledge are true and that all statements made on information and belief are believed to be true; and further that these statements were made with knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under section 1001 of Title 18 of the United States Code and that such willful false statements may jeopardize the validity of the application or any patent issued thereon.

Signed this 29th day of August , 2007

Tadaharu Isaka

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